Jan Please SEARCH REQUEST FORM 69962

Scientific and Technical Information Center

Requester's Full Name: S. Kumar Examiner #: 69594 Date: 7/1/02
Art Unit: 1621 Phone Number 308 4519 Serial Number: 09/887,933
Mail Box and Bldg/Room Location: CM1 7A07 7E12 Results Format Preferred (circle): PAPER DISK E-MAIL

If more than one search is submitted, please prioritize search s in order of need.

****

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Process for racemising an enantiomer-enriched Schiff base...

Inventors (please provide full names): Robert Patrick Hof et. al.

Earliest Priority Filing Date: 6/22/00

12. (Amended) A process for racemising an enantiomer-enriched Schiff base of a primary amide of an amino acid which process comprises contacting said enantiomer-enriched B1 Schiff base with a strong base in an organic solvent,

wherein said strong base is chemically reactive with water.

22. (Amended) The process of claim 12 wherein said enantiomer-enriched Schiff B2 base has been prepared from the primary amide of the amino acid in said organic solvent.

water.

13. (new) The process of claim 12 wherein the strong base is a metal alkoxide, a metal alkyl, a metal amide, or a metal hydride.

A1 14. (new) The process of claim 13 wherein the strong base is a metal alkoxide.

15. (new) The process of claim 12 wherein the strong base is present in an amount of 0.001-1000 mole% relative to the enantiomer-enriched Schiff base.

16. (new) The process of claim 15 wherein the strong base is present in an amount of 0.1-100 mole% relative to the enantiomer-enriched Schiff base.

17. (new) The process of claim 12 wherein the enantiomer-enriched Schiff base is an N-benzylidene primary amino acid amide.

18. (new) The process of claim 12 wherein the enantiomer-enriched Schiff base is derived from an aliphatic primary amino acid amide.

19. (new) The process of claim 18 wherein the enantiomer-enriched Schiff base is derived from tertiary-leucine amide.

20. (new) The process of claim 12 wherein the organic solvent is an aromatic hydrocarbon, a cyclic aliphatic hydrocarbon or an ether.

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jan.delaval@uspto.gov

Serial No. Not yet assigned
Docket No. 24615201xx00

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FILE COVERS 1907 - 8 Jul 2002 VOL 137 ISS 2
FILE LAST UPDATED: 7 Jul 2002 (20020707/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

CAS roles have been modified effective December 16, 2001. Please check your SDI profiles to see if they need to be revised. For information on CAS roles, enter HELP ROLES at an arrow prompt or use the CAS Roles thesaurus (/RL field) in this file.

=> d all tot l56

L56 ANSWER 1 OF 8 HCAPLUS COPYRIGHT 2002 ACS
AN **2001:936109** HCAPLUS
DN **136:54022**
TI Process for **racemizing** an **enantiomer**-enriched **Schiff base** of an **amino acid** amide using strong **bases**
IN **Hof, Robert Patrick; Hermsen, Petrus Johannes; De Bode, Ronus**
PA Neth.
SO U.S. Pat. Appl. Publ., 3 pp.
CODEN: USXXCO
DT Patent
LA English
IC ICM C07C251-02
NCL 564225000
CC 34-2 (**Amino Acids, Peptides,** and **Proteins**)
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	US 2001056209	A1	20011227	US 2001-887933	20010622
	NL 1015495	C2	20011228	NL 2000-1015495	20000622
	EP 1167347	A1	20020102	EP 2001-202359	20010621
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
	JP 2002037767	A2	20020206	JP 2001-190159	20010622
PRAI	NL 2000-1015495	A	20000622		

AB The invention relates to a process for **racemizing** an **enantiomer**-enriched **Schiff base** of a primary **amino acid amide** with a strong **base** that is chem. reactive towards water. The reaction is conducted in an org. solvent (e.g., THF). Preferably a **metal alkoxide**, a **metal alkyl**, a **metal amide**, or

a **metal hydride**, in particular a **metal alkoxide** (e.g., KOCMe3) is applied as the strong **base**. As the **Schiff base** preferably N-benzylidene primary **amino acid amide** (e.g., N-benzylidene-(R)-tertiary-leucine **amide**) is used, with the primary **amino acid amide** preferably being derived from an aliph. primary **amino acid amide**, for example tertiary-leucine **amide**. As org. solvent use is preferably made of an arom. hydrocarbon, a cyclic, aliph. hydrocarbon or a ether, in particular an arom. hydrocarbon is applied. The invention may also be applied for the **racemization** of an **enantiomer**-enriched primary **amino acid amide**.

ST **racemization Schiff base amino acid** amide; benzylideneleucine amide **base racemization**

IT **Amides**, processes
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(amino, **Schiff bases**; process for **racemizing** an **enantiomer**-enriched **Schiff base** of an **amino acid** amide using strong **bases**)

IT **Hydrides**
Metal alkoxides
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(**bases**; process for **racemizing** an **enantiomer**-enriched **Schiff base** of an **amino acid amide** using strong **bases**)

IT **Schiff bases**
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(of an **amino acid** amide; process for **racemizing** an **enantiomer**-enriched **Schiff base** of an **amino acid** amide using strong **bases**)

IT **Racemization**
(process for **racemizing** an **enantiomer**-enriched **Schiff base** of an **amino acid** amide using strong **bases**)

IT **Bases, processes**
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(process for **racemizing** an **enantiomer**-enriched **Schiff base** of an **amino acid** amide using strong **bases**)

IT Aromatic hydrocarbons, uses
Cycloalkanes
RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PYP (Physical process); REM (Removal or disposal); PROC (Process); USES (Uses)
(solvents; process for **racemizing** an **enantiomer**-enriched **Schiff base** of an **amino acid** amide using strong **bases**)

IT **124-41-4**, Sodium methoxide **141-52-6**, Sodium ethoxide 865-47-4 381724-98-7
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(process for **racemizing** an **enantiomer**-enriched **Schiff base** of an **amino acid** amide using strong **bases**)

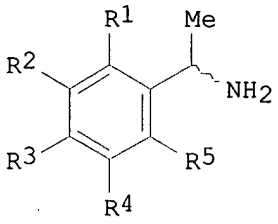
IT 381724-99-8P

RL: SPN (Synthetic preparation); PREP (Preparation)
(process for **racemizing** an **enantiomer**-enriched **Schiff base** of an **amino acid** amide using strong **bases**)

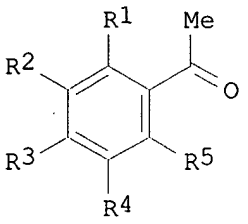
L56 ANSWER 2 OF 8 HCAPLUS COPYRIGHT 2002 ACS
AN 1998:79714 HCAPLUS
DN 128:167308
TI Method for producing **racemic** phenethylamines
IN Stelzer, Uwe
PA Bayer A.-G., Germany
SO Ger. Offen., 12 pp.
CODEN: GWXXBX
DT Patent
LA German
IC ICM C07C211-29
ICS C07C211-27; C07C209-84; C07B055-00
ICA C07C251-16
CC 26-9 (Biomolecules and Their Synthetic Analogs)
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 19629692	A1	19980129	DE 1996-19629692	19960723
	WO 9803465	A1	19980129	WO 1997-EP3691	19970711
	W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
	RW: GH, KE, LS, MW, SD, SZ, UG, ZW, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
	AU 9736223	A1	19980210	AU 1997-36223	19970711
	EP 923534	A1	19990623	EP 1997-932809	19970711
	EP 923534	B1	20001004		
	R: BE, CH, DE, DK, ES, FR, GB, IT, LI, NL				
	BR 9710391	A	19990817	BR 1997-10391	19970711
	CN 1226228	A	19990818	CN 1997-196707	19970711
	JP 2000514813	T2	20001107	JP 1998-506503	19970711
	ES 2150267	T3	20001116	ES 1997-932809	19970711
	US 6046351	A	20000404	US 1999-230232	19990119
PRAI	DE 1996-19629692	A	19960723		
	WO 1997-EP3691	W	19970711		

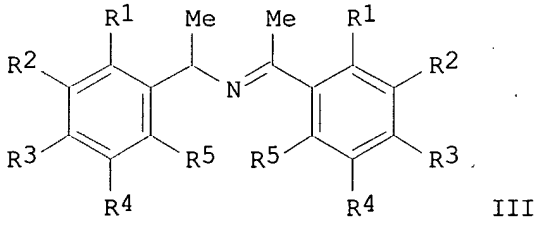
OS CASREACT 128:167308; MARPAT 128:167308
GI



I



II



III

AB **Racemic** phenethylamines I [R1-R5 = H, halo, cyano, nitro, **alkyl**, alkoxy, alkylthio, alkylsulfinyl, etc.] are prepd. by condensing their optically active stereoisomers with acetophenone derivs. II, treating the resulting optically active **Schiff base** [optically active III] with **metal** hydroxide contg. 0.1-50% water, and treating the resulting **racemic Schiff base** with acid in the presence of water. Thus, (S)-1-(4-chlorophenyl)ethylamine was treated with 4-chloroacetophenone in toluene contg. tetra-Bu orthotitanate at room temp. followed by refluxing 6 h to give 91% the corresponding (S) **Schiff base**, which was stirred with KOH contg. 15 wt.% water for 16 h and then heated at 130-160.degree. followed by cooling and refluxing with 2N aq. H2SO4 for 2 h to give the title compd. (.+-.)-1-(4-chlorophenyl)ethylamine.

ST **racemic** phenethylamine prepn

IT 202827-93-8P
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(method for producing **racemic** phenethylamines)

IT 99-91-2 4187-56-8, (S)-1-(4-Chlorophenyl)ethylamine 6299-02-1
RL: RCT (Reactant); RACT (Reactant or reagent)
(method for producing **racemic** phenethylamines)

L56 ANSWER 3 OF 8 HCAPLUS COPYRIGHT 2002 ACS

AN **1996:231688** HCAPLUS

DN **124:288970**

TI **Racemization** of optically active .alpha.-arylalkylamines

IN Tsucha, Toyohito; Sugyama, Naoko; Takemoto, Tadashi

PA Ajinomoto Kk, Japan

SO Jpn. Kokai Tokkyo Koho, 4 pp.
CODEN: JKXXAF

DT Patent

LA Japanese

IC ICM C07C211-27
ICS C07C209-88

CC 25-4 (Benzene, Its Derivatives, and Condensed Benzenoid Compounds)

FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 08027073	A2	19960130	JP 1994-171190	19940722

OS MARPAT 124:288970

AB Optically active **Schiff bases** formed from optically active ArCHRNH2 (I; Ar = aryl; R = alkyl) and arylaldehydes are treated

with **bases** and the resulting **racemized Schiff bases** are hydrolyzed to give **racemic** I. The obtained **racemates** are useful as materials for resoln. to obtain isomers useful as resolving agents and intermediates for sweet substances. (S)-.alpha.-phenylpropylamine [(S)-I] and p-ClC6H4CHO were dissolved in CH2Cl2 and the soln. was treated with MgSO4 under stirring overnight to give (S)-N-(p-chlorobenzylidene)-.alpha.-phenylpropylamine. The **Schiff base** dissolved in Me3COH was treated with Me3COK under reflux for 5 h, followed by treatment of the reaction product with HCl at room temp. for 30 min to give (.+-.)-I at **racemization** rate 90.5%.

ST arylalkylamine **Schiff base racemization** hydrolysis; **racemic** arylalkylamine prepn

IT **Racemization**
(**racemization** of .alpha.-arylalkylamines by treatment of **Schiff bases** of the isomers and arylaldehydes with **bases** followed by hydrolysis)

IT **Amines**, reactions
Hydroxides
RL: RCT (Reactant)
(**racemization** of .alpha.-arylalkylamines by treatment of **Schiff bases** of the isomers and arylaldehydes with **bases** followed by hydrolysis)

IT **Schiff bases**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(**racemization** of .alpha.-arylalkylamines by treatment of **Schiff bases** of the isomers and arylaldehydes with **bases** followed by hydrolysis)

IT **Amines**, preparation
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(.alpha.-arylalkyl; **racemization** of .alpha.-arylalkylamines by treatment of **Schiff bases** of the isomers and arylaldehydes with **bases** followed by hydrolysis)

IT **Alcohols, reactions**
RL: RCT (Reactant)
(**metal salts, racemization** of .alpha.-arylalkylamines by treatment of **Schiff bases** of the isomers and arylaldehydes with **bases** followed by hydrolysis)

IT 98-84-0P, .alpha.-Phenylethylamine 2941-20-0P, .alpha.-Phenylpropylamine
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)
(**racemization** of .alpha.-arylalkylamines by treatment of **Schiff bases** of the isomers and arylaldehydes with **bases** followed by hydrolysis)

IT 104-88-1, p-Chlorobenzaldehyde, reactions 865-47-4, Potassium tert-butoxide 1310-58-3, Potassium hydroxide, reactions 3082-64-2 3789-59-1, (S)-.alpha.-Phenylpropylamine 4187-48-8 6674-22-2, DBU 74879-38-2 74879-40-6 175842-06-5
RL: RCT (Reactant)
(**racemization** of .alpha.-arylalkylamines by treatment of **Schiff bases** of the isomers and arylaldehydes with **bases** followed by hydrolysis)

IT 175842-05-4P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(**racemization** of .alpha.-arylalkylamines by treatment of **Schiff bases** of the isomers and arylaldehydes with **bases** followed by hydrolysis)

L56 ANSWER 4 OF 8 HCAPLUS COPYRIGHT 2002 ACS
AN **1992:42063** HCAPLUS
DN **116:42063**

TI Process for **racemization** of optically active **amino acid** amides

IN Boesten, Wilhelmus Hubertus Joseph

PA Stamicarbon B. V., Neth.

SO Eur. Pat. Appl., 9 pp.
CODEN: EPXXDW

DT Patent

LA English

IC ICM C07C237-20
ICS C07C231-20; C07B055-00

CC 34-2 (**Amino Acids**, **Peptides**, and **Proteins**)

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 442585	A1	19910821	EP 1991-200307	19910214
	EP 442585	B1	19940720		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, NL, SE				
	NL 9000387	A	19910916	NL 1990-387	19900216
	HU 56531	A2	19910930	HU 1991-481	19910213
	HU 212704	B	19961028		
	ES 2061155	T3	19941201	ES 1991-200307	19910214
	JP 07070027	A2	19950314	JP 1991-22138	19910215
	JP 2941444	B2	19990825		
	CZ 280920	B6	19960515	CZ 1991-417	19910218
PRAI	NL 1990-387		19900216		

AB Optically active **amino acid** amides are **racemized** by a process comprising conversion of the optically active amide or its **Schiff base** in the presence of 0.5-4 equiv of an aldehyde to its addn. salt at 75-100.degree. using 1-2 equiv of a **racemic** carboxylic acid with the addn. of 0.5-3 equiv of H2O. No aldehyde is needed when the **Schiff base** is the starting material. Thus, 0-10 mol D-N-benzylidenephenylglycineamide, 0.10 mol DL-mandelic acid, 200 mL PhMe, 50 mL EtOAc, and 0.15 mol H2O were stirred 4 h at 85.degree.. The **Schiff base** addn. salt formed was filtered and hydrolyzed by 6N HCl to give DL-phenylglycineamide.HCl. The above reaction carried out without addn. of water gave only 12.2 g of intermediate **Schiff base** addn. salt, compared to 29.9 g when H2O was added.

ST chiral **amino acid** amide **racemization**; **racemic amino acid** amide prepn; benzylidene phenylglycineamide prepn **racemization**

IT **Racemization**
(of optically active **amino acid** amides via **Schiff bases**)

IT **Schiff bases**
RL: RCT (Reactant)
(**amino acid**, formation and **racemization** of, in prepn. of **racemic amino acid** amides)

IT **Amides**, preparation
RL: SPN (Synthetic preparation); PREP (Preparation)
(amino, **racemic**, prepn. of, via **racemization** of optically active **Schiff base** derivs.)

IT 100-52-7P, Benzaldehyde, preparation
RL: PREP (Preparation)
(**Schiff base** formation of, with optically active **amino acid** amides, in **racemization** reaction)

IT 78-84-2 89-98-5, o-Chlorobenzaldehyde 50984-52-6, Anisaldehyde
RL: PROC (Process)
(**Schiff base** formation of, with **racemic amino acid** amides)

IT 138228-63-4P 138258-73-8P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(prepn. and decompn. of)
IT 138228-56-5P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(prepn. and decompn. of, in prepn. of **racemic amino acid** amide)
IT 54397-23-8P 60079-51-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)
IT 51703-58-3P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of, via **racemization** of corresponding optically active **Schiff base**)
IT 700-63-0 4726-84-5 19298-72-7 67412-95-7 108888-96-6 108888-97-7
108888-98-8 108888-99-9 138228-57-6 138228-58-7 138228-59-8
138228-60-1 138228-61-2 138258-70-5 138258-71-6
RL: RCT (Reactant)
(**racemization** of)
IT 6485-67-2
RL: RCT (Reactant)
(**racemization** of, via **Schiff base**)
IT 58429-87-1 72151-95-2
RL: PROC (Process)
(resoln. of, via **Schiff base**)
IT 64-19-7, Acetic acid, reactions 611-72-3, DL-Mandelic acid
RL: RCT (Reactant)
(salification of, with optically active **amino acid** amide **Schiff bases**)
IT 611-71-2, D-Mandelic acid 17199-29-0, L-Mandelic acid
RL: PROC (Process)
(salt formation of, with **racemic amino acid** amide **Schiff bases**)

L56 ANSWER 5 OF 8 HCAPLUS COPYRIGHT 2002 ACS
AN **1992:42062** HCAPLUS
DN **116:42062**
TI Preparation of optically active **amino acid** amides via **Schiff base** salts.
IN Boesten, Wilhelmus Hubertus Joseph
PA Stamicarbon B. V., Neth.
SO Eur. Pat. Appl., 12 pp.
CODEN: EPXXDW
DT Patent
LA English
IC ICM C07C231-20
ICS C07C237-20; C07C249-02; C07B057-00
CC 34-2 (**Amino Acids, Peptides,** and **Proteins**)
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 442584	A1	19910821	EP 1991-200306	19910214
	EP 442584	B1	19931110		
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, NL, SE				
	NL 9000386	A	19910916	NL 1990-386	19900216
	HU 56532	A2	19910930	HU 1991-482	19910213
	HU 212703	B	19961028		
	AT 97125	E	19931115	AT 1991-200306	19910214
	ES 2062660	T3	19941216	ES 1991-200306	19910214
	JP 05178805	A2	19930720	JP 1991-22137	19910215
	JP 2854148	B2	19990203		
	US 5306826	A	19940426	US 1991-655623	19910215

CZ 281203 B6 19960717 CZ 1991-418 19910218
PRAI NL 1990-386 19900216
EP 1991-200306 19910214
AB Title compds. are prepd. from their **racemic** mixts. by conversion of the mixts. to **Schiff base** salts with optically active carboxylic acids in a process using 0.5-4 equiv aldehyde and 0.5-3 equiv H2O, followed by hydrolysis. Thus, a mixt. of 0.10 mL DL-phenylglycine amide, 0.10 mol D-mandelic acid, 230 mL PhMe, 20 mL PhCHO, and 0.10 mol H2O was stirred for 2 h at 88.degree.. After cooling, the **Schiff base** addn. salt was filtered and hydrolyzed by 6N HCl to give L-phenylglycine amide.HCl. Resoln. was also accomplished starting with the **Schiff base** of the **amino acid** amides.
ST chiral **amino acid** amide prepn; resoln **racemic amino acid** amide; benzylidene phenylglycineamide chiral resoln
IT **Schiff bases**
RL: FORM (Formation, nonpreparative)
(formation of, in resoln. of **amino acid** amides)
IT Resolution
(of **amino acid** amides via **Schiff bases**)
IT **Amides**, preparation
RL: SPN (Synthetic preparation); PREP (Preparation)
(amino, chiral, prepn. of, via resoln. of corresponding **Schiff base racemic** mixts.)
IT 78-84-2, Isobutyraldehyde 89-98-5, o-Chlorobenzaldehyde 50984-52-6, Anisaldehyde
RL: PROC (Process)
(**Schiff base** formation of, with **racemic amino acid** amides, in prepn. of optically active **amino acid** amides)
IT 100-52-7P, Benzaldehyde, preparation
RL: SPN (Synthetic preparation); PREP (Preparation)
(**Schiff base** formation of, with **racemic amino acid** amides, in prepn. of optically active **amino acid** amides)
IT 138228-65-6P 138228-66-7P 138228-68-9P 138228-69-0P 138258-73-8P 138258-74-9P 138258-75-0P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(prepn. and decompn. of, in prepn. of optically active **amino acid** amides)
IT 875-74-1P, D-Phenylglycine 2935-35-5P, L-Phenylglycine 16120-92-6P, L-Methionineamide hydrochloride 32462-30-9P, L-p-Hydroxyphenylglycine 53958-19-3P 54397-23-8P 60079-51-8P 63291-39-4P 82795-51-5P 138228-64-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of, from **racemate**, via **Schiff base** salt with optically active carboxylic acid)
IT 108945-11-5 108945-13-7 138258-76-1
RL: RCT (Reactant)
(resoln. and hydrolysis of, optically active **amino acid** amides from)
IT 4510-08-1
RL: PROC (Process)
(resoln. of, via **Schiff base**)
IT 58429-87-1, DL-Phenylglycineamide 72151-95-2
RL: PROC (Process)
(resoln. of, via **Schiff base** with benzaldehyde)
IT 98-79-3, L-2-Pyrrolidone-5-carboxylic acid 611-71-2, D-Mandelic acid 1152-61-0 17199-29-0, L-Mandelic acid
RL: PROC (Process)
(salt formation of, with **racemic amino acid**

amide **Schiff bases**, in prepn. of optically active **amino acid** amides)

L56 ANSWER 6 OF 8 HCAPLUS COPYRIGHT 2002 ACS
AN 1986:109498 HCAPLUS
DN 104:109498
TI Optically active .alpha.-amino-.epsilon.-caprolactam
IN Markowicz, Stanislaw; Leplawy, Miroslaw; Witkowski, Kazimierz; Kociolek, Karol; Kuswik, Gabriela; Krawczyk, Henryk; Lewandowska, Ewa; Olejniczak, Bogdan
PA Politechnika Lodzka, Pol.
SO Pol., 2 pp.
CODEN: POXXA7
DT Patent
LA Polish
IC C07D223-10
CC 27-21 (Heterocyclic Compounds (One Hetero Atom))
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PL 124435	B2	19830131	PL 1980-228617	19801218

AB Optically active .alpha.-amino-.epsilon.-caprolactam (I) is prepd. by contacting **racemic** I with an optically active terpene aldehyde or ketone in presence of BF3 etherate or p-toluenesulfonic acid (II) (as a catalyst) in an org. solvent. The **Schiff base** obtained is reacted with BuLi in presence of (Me2CH)2NH, or with a **metal hydride** in THF or ether. The mixt. was treated with an aq. mineral acid, and the product was sepd. Thus, **racemic** I was resolved by treatment with (+)-mytenal, II, BuLi, (Me2CH)2NH and HCl to give I.HCl, [.alpha.]20D = -6.3.degree..
ST aminocaprolactam resoln; caprolactam amino resoln
IT 100325-27-7P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(prepn. and hydrolysis of)
IT 26081-07-2P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)
IT 17929-90-7
RL: PROC (Process)
(resoln. of)

L56 ANSWER 7 OF 8 HCAPLUS COPYRIGHT 2002 ACS
AN 1982:598518 HCAPLUS
DN 97:198518
TI **Deracemization** by enantioselective protonation. Application to an .alpha.-amino acid, phenylglycine
AU Duhamel, Lucette; Plaquevent, Jean Christophe
CS Lab. Chim. Org., Fac. Sci. Tech. Rouen, Mont Saint-Aignan, F-76130, Fr.
SO Bull. Soc. Chim. Fr. (1982), (3-4, Pt. 2), 75-83
CODEN: BSCFAS; ISSN: 0037-8968
DT Journal
LA French
CC 34-2 (Amino Acids, Peptides, and Proteins)
Section cross-reference(s): 22
AB Phenylglycine esters were converted into **Schiff bases**, **metalated** by a Li **amide**, and then protonated by a chiral acid to give optically active starting materials (enantiomer excess as high as 70%). Chiral acids can easily be retrieved after protonation with excellent yields and conservation of enantiomeric purity. A mechanism responsible for the asym. induction is suggested by means of a study of the parameters modifying the selectivity, such as the nature of protecting groups, chiral acid, and lithium **amide**.
ST resoln phenylglycine enantioselective protonation; substituent effect

benzylidenephenylglycinate resoln
IT Asymmetric synthesis and induction
(of benzylidenephenylglycine ester by enantioselective protonation)
IT Resolution
(of benzylidenephenylglycine esters)
IT Substituent effect
(on resoln. of benzylidenephenylglycinate by enantioselective protonation)
IT Protonation and Proton transfer reaction
(enantioselective, of lithiated benzylidenephenylglycinate)
IT 3886-69-9
RL: RCT (Reactant)
(acylation of)
IT 74842-56-1 76769-54-5 76769-56-7 76821-61-9
RL: RCT (Reactant)
(benzylidenephenylglycinate resoln. in presence of)
IT 2835-06-5
RL: RCT (Reactant)
(esterification of)
IT 816-43-3 4111-54-0 4111-55-1 38227-87-1
RL: RCT (Reactant)
(metalation by, of benzylidenephenylglycinate)
IT 5933-40-4P 70811-66-4P 76821-62-0P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation)
(prepn. and hydride redn. of)
IT 43189-03-3P 43189-47-5P 63430-99-9P 83529-43-5P 83529-44-6P
83529-45-7P 83529-46-8P 83529-47-9P 83572-72-9P 83572-73-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. and resoln. of, by enantioselective protonation)
IT 15028-40-7P 19883-41-1P 36123-72-5P 39251-36-0P 55130-90-0P
59410-82-1P 72651-17-3P 83529-48-0P 83529-49-1P 83529-50-4P
83529-51-5P 83572-23-0P 83572-24-1P 83572-25-2P 83572-26-3P
83572-27-4P 83572-28-5P 83572-29-6P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)
IT 63903-05-9P 68906-71-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of, by resoln. via enantioselective protonation)
IT 2743-38-6 5123-55-7 17199-29-0 17257-71-5 51591-38-9 65259-81-6
65259-82-7 68870-86-0 68870-87-1 68870-88-2 68870-89-3
68870-90-6 68870-91-7 68870-92-8 74817-66-6 74817-67-7
74817-68-8 74817-69-9 74817-72-4 83529-37-7 83529-38-8
83529-39-9 83529-40-2 83529-41-3 83529-42-4
RL: RCT (Reactant)
(protonation by, of lithiated benzylidenephenylglycinate)
IT 76769-55-6 76821-63-1 76821-64-2
RL: RCT (Reactant)
(protonation by, of lithiated benzylidenephenylgycinate)
IT 100-10-7 100-52-7, reactions 104-87-0 104-88-1, reactions 105-07-7
123-11-5, reactions 135-02-4 591-31-1
RL: RCT (Reactant)
(reaction of, with phenylglycine ester)

L56 ANSWER 8 OF 8 HCAPLUS COPYRIGHT 2002 ACS
AN 1981:121892 HCAPLUS
DN 94:121892
TI **Deracemization** by **enantioselective** protonation. IV.
An improved method for the **enantiomeric** enrichment of .alpha.-**amino acids** using **metalation** by means of chiral **amides**
AU Duhamel, Lucette; Plaquevent, Jean Christophe
CS Lab. Chim. Org., Fac. Sci. Tech. Rouen, Mont Saint Aignan, 76130, Fr.
SO Tetrahedron Lett. (1980), 21(26), 2521-4

CODEN: TELEAY; ISSN: 0040-4039
DT Journal
LA English
CC **34-2** (Synthesis of **Amino Acids**, **Peptides**, and **Proteins**)
AB Optically active .alpha.-**amino acid** esters were prepd. by **metalation** of the corresponding **Schiff bases** by chiral lithium **amides** followed by protonation by an achiral or a chiral acid. Thus, PhCH:NCHPhCO2Me underwent sequential **metalation** with (R)-PhCHMeNRLi (R = Me, Et, Pr) (-50.degree.), reaction with (2R,3R)-[HO2CCHO2C(CMe3)]2 (-70.degree.) in the presence of (R)-PhCHMeNHR (R as before), and hydrolysis to give PhCH(NH3Cl)CO2Me with **enantiomeric** excess of 70%.
ST **enantioselective** protonation **amino acid** **deracemization**
IT **Amino acids**, reactions
RL: RCT (Reactant)
(**deracemization** of, by **enantioselective** protonation)
IT Resolution
(of **amino acids** by **enantioselective** protonation)
IT **Racemization**
(de-, of **amino acids** by **enantioselective** protonation)
IT Protonation and Proton transfer reaction
(**enantioselective**, in **deracemization** of **amino acids**)
IT 43189-47-5
RL: RCT (Reactant)
(**deracemization** of)
IT 65259-81-6 68870-92-8 76769-55-6 76821-63-1
RL: RCT (Reactant)
(**enantioselective** protonation by, of metalated enolate of benzylidenephenylglycine Me ester)
IT 63903-05-9
RL: RCT (Reactant)
(**enantioselective** protonation of)
IT 74842-56-1 76769-54-5 76769-56-7 76821-61-9
RL: RCT (Reactant)
(metalation by, of **Schiff base**)
IT 15028-39-4P 19883-41-1P
RL: SPN (Synthetic preparation); PREP (Preparation)
(prepn. of)
IT 76821-64-2
RL: RCT (Reactant)
(protonation by, of metalated enolate of benzylidenephenylglycine Me ester)
IT 5933-40-4 70811-66-4 76821-62-0
RL: RCT (Reactant)
(reaction of, with metalated **Schiff base** and tartaric acid esters)

=> fil reg
FILE 'REGISTRY' ENTERED AT 12:40:17 ON 08 JUL 2002
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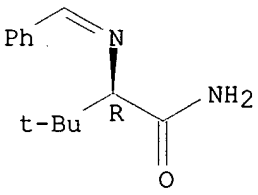
http://www.cas.org/ONLINE/STN/STNOTES/stnotes27.pdf

=> d ide can l58

L58 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2002 ACS
RN **381724-98-7** REGISTRY
CN Butanamide, 3,3-dimethyl-2-[(phenylmethylene)amino]-, (2R)- (9CI) (CA INDEX NAME)
FS STEREOSEARCH
MF C13 H18 N2 O
SR CA
LC STN Files: CA, CAPLUS, USPATFULL

Absolute stereochemistry.
Double bond geometry unknown.



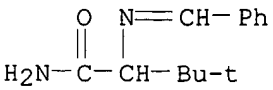
PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

1 REFERENCES IN FILE CA (1967 TO DATE)
1 REFERENCES IN FILE CAPLUS (1967 TO DATE)

REFERENCE 1: 136:54022

=> d ide can l59

L59 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2002 ACS
RN **381724-99-8** REGISTRY
CN Butanamide, 3,3-dimethyl-2-[(phenylmethylene)amino]- (9CI) (CA INDEX NAME)
FS 3D CONCORD
MF C13 H18 N2 O
SR CA
LC STN Files: CA, CAPLUS, USPATFULL



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

1 REFERENCES IN FILE CA (1967 TO DATE)
1 REFERENCES IN FILE CAPLUS (1967 TO DATE)

REFERENCE 1: 136:54022

=> fil wpix
FILE 'WPIX' ENTERED AT 12:53:36 ON 08 JUL 2002
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FILE LAST UPDATED: 04 JUL 2002 <20020704/UP>
MOST RECENT DERWENT UPDATE 200242 <200242/DW>
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>>> SLART (Simultaneous Left and Right Truncation) is now
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/BIX is also provided which comprises both /BI and /ABEX <<<

>>> Update 2002-42 does not contain any new polymer indexing <<<

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enabled in WPINDEX/WPIDS and WPIX >>>

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http://www.derwent.com/userguides/dwpi_guide.html <<<

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L71 ANSWER 1 OF 10 WPIX (C) 2002 THOMSON DERWENT
AN 2002-194879 [25] WPIX
DNC C2002-060158
TI **Racemization** process involves providing organic solvent used for **racemizing** enantiomer-enriched **Schiff** base of primary amino acid amide with strong base that is chemically reactive towards water.
DC E16
IN DE BODE, R; HERMSEN, P J; HOF, R P
PA (STAM) DSM NV; (DBOD-I) DE BODE R; (HERM-I) HERMSEN P J; (HOFR-I) HOF R P
CYC 28
PI US 2001056209 A1 20011227 (200225)* 3p C07C251-02
EP 1167347 A1 20020102 (200225) EN C07C249-02 <--
R: AL AT BE CH CY DE DK ES FI FR GB GR IE IT LI LT LU LV MC MK NL PT RO SE SI TR
NL 1015495 C2 20011228 (200225) C07C249-02
JP 2002037767 A 20020206 (200226) 12p C07C249-02
ADT US 2001056209 A1 US 2001-887933 20010622; EP 1167347 A1 EP 2001-202359 20010621; NL 1015495 C2 NL 2000-1015495 20000622; JP 2002037767 A JP 2001-190159 20010622
PRAI NL 2000-1015495 20000622
IC ICM C07C249-02; C07C251-02
ICS C07C237-00; C07C251-16; C07C251-24

ICA **C07B055-00**
AB US2001056209 A UPAB: 20020418
NOVELTY - A strong base that is chemically reactive towards water is used in an organic solvent for **racemizing** an enantiomer-enriched **Schiff** base of a primary amino acid amide.
USE - For enantiomer-enriched primary amino acid amide.
ADVANTAGE - Allows enantiomer-enriched **Schiff** bases of primary amino acid amides to be **racemized** efficiently, with strongly reduced likelihood of byproducts being formed.
Dwg.0/0
FS CPI
FA AB; DCN
MC CPI: E10-A20B

L71 ANSWER 2 OF 10 WPIX (C) 2002 THOMSON DERWENT
AN 1998-101789 [10] WPIX
DNC C1998-033669
TI Preparation of **racemic** phenylethyl-amine derivatives - by reaction of optically-active amine with identically ring-substituted acetophenone to give **Schiff** base, **racemisation** and final cleavage.
DC B05
IN STELZER, U
PA (FARB) BAYER AG
CYC 77
PI DE 19629692 A1 19980129 (199810)* 11p C07C211-29
WO 9803465 A1 19980129 (199811) C07C209-68
RW: AT BE CH DE DK EA ES FI FR GB GH GR IE IT KE LS LU MC MW NL OA PT SD SE SZ UG ZW
W: AL AM AT AU AZ BA BB BG BR BY CA CH CN CU CZ DE DK EE ES FI GB GE HU IL IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MD MG MK MN MW MX NO NZ PL PT RO RU SD SE SG SI SK TJ TM TR TT UA UG US UZ VN
AU 9736223 A 19980210 (199827) C07C209-68
EP 923534 A1 19990623 (199929) DE C07C209-68
R: BE CH DE DK ES FR GB IT LI NL
CN 1226228 A 19990818 (199951) C07C209-68
BR 9710391 A 19990817 (199954) C07C209-68
HU 9903251 A2 20000128 (200015) C07C209-68
US 6046351 A 20000404 (200024) C07C305-04
EP 923534 B1 20001004 (200050) DE C07C209-68
R: BE CH DE DK ES FR GB IT LI NL
DE 59702432 G 20001109 (200059) C07C209-68
JP 2000514813 W 20001107 (200059) 26p C07C209-68
MX 9900880 A1 19990801 (200063) C07C209-66
ES 2150267 T3 20001116 (200064) C07C209-68
KR 2000067873 A 20001125 (200130) C07C209-68
IL 127970 A 20010826 (200157) C07C211-27
MX 204324 B 20010919 (200239) C07B055-00 <--
ADT DE 19629692 A1 DE 1996-19629692 19960723; WO 9803465 A1 WO 1997-EP3691 19970711; AU 9736223 A AU 1997-36223 19970711; EP 923534 A1 EP 1997-932809 19970711, WO 1997-EP3691 19970711; CN 1226228 A CN 1997-196707 19970711; BR 9710391 A BR 1997-10391 19970711, WO 1997-EP3691 19970711; HU 9903251 A2 WO 1997-EP3691 19970711, HU 1999-3251 19970711; US 6046351 A WO 1997-EP3691 19970711, US 1999-230232 19990119; EP 923534 B1 EP 1997-932809 19970711, WO 1997-EP3691 19970711; DE 59702432 G DE 1997-502432 19970711, EP 1997-932809 19970711, WO 1997-EP3691 19970711; JP 2000514813 W WO 1997-EP3691 19970711, JP 1998-506503 19970711; MX 9900880 A1 MX 1999-880 19990122; ES 2150267 T3 EP 1997-932809 19970711; KR 2000067873 A WO 1997-EP3691 19970711, KR 1999-700258 19990115; IL 127970 A IL 1997-127970 19970711; MX 204324 B MX 1999-880 19990122
FDT AU 9736223 A Based on WO 9803465; EP 923534 A1 Based on WO 9803465; BR 9710391 A Based on WO 9803465; HU 9903251 A2 Based on WO 9803465; US 6046351 A Based on WO 9803465; EP 923534 B1 Based on WO 9803465; DE

59702432 G Based on EP 923534, Based on WO 9803465; JP 2000514813 W Based on WO 9803465; ES 2150267 T3 Based on EP 923534; KR 2000067873 A Based on WO 9803465; IL 127970 A Based on WO 9803465
PRAI DE 1996-19629692 19960723
IC ICM C07C209-66; C07C209-68; C07C211-27; C07C211-29; C07C305-04
ICS C07C209-84; C07C211-03; C07C217-544; C07C255-49; C07C255-50; C07C313-12; C07C317-14; C07C323-32
ICA **C07B055-00**
AB DE 19629692 A UPAB: 19980309
Preparation of **racemic** phenylethylamine derivatives of formula (I) by:
(a) reacting optically-active (I) with an acetophenone derivative of formula (II), where the phenyl substitution in (I) and (II) is identical, optionally in the presence of a solvent and/or catalyst;
(b) reacting the optically-active **Schiff** base (III) with a metal hydroxide and water content = 0.1-50 wt.%, optionally under an inert atmosphere, and
(c) treating the obtained **racemic Schiff** bases with aqueous acid.
R1-R5 = H, halo, cyano, nitro, alkyl, alkoxy, alkylthio, alkylsulphinyl, alkylsulphonyl, dialkylamino, haloalkyl, haloalkoxy, haloalkylthio, haloalkylsulphinyl or haloalkylsulphonyl)
ADVANTAGE - The method affords a high degree of **racemisation**.
Dwg.0/0
FS CPI
FA AB; GI; DCN
MC CPI: B10-A10; B10-A15; B10-B01A; B10-B04B

L71 ANSWER 3 OF 10 WPIX (C) 2002 THOMSON DERWENT
AN **1996-136245** [14] WPIX
DNC **C1996-042431**
TI **Racemisation** of optically active alpha-aryl-alkylamine - by **racemising** optically active **Schiff** base of alpha-aryl-alkylamine and aryl-aldehyde with base, then hydrolysing.
DC B05 E14
PA (AJIN) AJINOMOTO KK
CYC 1
PI JP 08027073 A 19960130 (199614)* 4p C07C211-27
ADT JP 08027073 A JP 1994-171190 19940722
PRAI JP 1994-171190 19940722
IC ICM C07C211-27
ICS C07C209-88
AB JP 08027073 A UPAB: 19960405
Racemisation of an optically active alpha-aryl-alkylamine comprises contacting an optically active **Schiff** base (prepd. from an optically active alpha-aryl-alkylamine of formula Ar-CHR-NH2 (I) and aryl-aldehyde) with a base to **racemise** and then hydrolysing the **Schiff** base. Ar = aryl; and R = alkyl.
USE - The optically active alpha-aryl-alkylamine is useful as an optically resolving agent for obtaining an optically active cpd. from **racemic** carboxylic acids. The s-isomer of the amine of formula (I; Ar = phenyl or methyl-substd. phenyl) is important as the starting material for a strongly sweet cpd..
ADVANTAGE - The method is carried out with safety with a cheap reagent.
Dwg.0/0
FS CPI
FA AB; DCN
MC CPI: B10-B04B; B11-B; E10-B04C; E11-J

L71 ANSWER 4 OF 10 WPIX (C) 2002 THOMSON DERWENT
AN 1991-247269 [34] WPIX

DNC C1991-107300
TI **Racemisation** of optically active aminoacid amide(s) - by reaction of amide with carboxylic acid in presence of aldehyde and water.
DC B05 E14
IN BOESTEN, W H; BOESTEN, W H J
PA (STAM) DSM NV; (STAM) STAMICARBON BV
CYC 19
PI EP 442585 A 19910821 (199134)*
R: AT BE CH DE ES FR GB GR IT LI NL SE
NL 9000387 A 19910916 (199140)
HU 56531 T 19910930 (199143)
CS 9100417 A 19910915 (199148)
EP 442585 B1 19940720 (199428) EN 10p C07C237-20
R: AT BE CH DE DK ES FR GB GR IT LI NL SE
DE 69102896 E 19940825 (199433) C07C237-20
ES 2061155 T3 19941201 (199504) C07C237-20
SG 9401335 A 19950113 (199513)
JP 07070027 A 19950314 (199519) 7p C07C237-04
CZ 280920 B6 19960515 (199627) C07C231-16
HU 212704 B 19961028 (199702) C07B055-00 <--
JP 2941444 B2 19990825 (199940) 7p C07C231-16
KR 167558 B1 19990320 (200042) C07C229-06
ADT EP 442585 A EP 1991-200307 19910214; NL 9000387 A NL 1990-387 19900216; EP 442585 B1 EP 1991-200307 19910214; DE 69102896 E DE 1991-602896 19910214, EP 1991-200307 19910214; ES 2061155 T3 EP 1991-200307 19910214; SG 9401335 A SG 1994-1335 19940921; JP 07070027 A JP 1991-22138 19910215; CZ 280920 B6 CS 1991-417 19910218; HU 212704 B HU 1991-481 19910213; JP 2941444 B2 JP 1991-22138 19910215; KR 167558 B1 KR 1991-2590 19910213
FDT DE 69102896 E Based on EP 442585; ES 2061155 T3 Based on EP 442585; SG 9401335 A Previous Publ. EP 442585; CZ 280920 B6 Previous Publ. CS 9100417; HU 212704 B Previous Publ. HU 56531; JP 2941444 B2 Previous Publ. JP 07070027
PRAI NL 1990-387 19900216
REP EP 199407; EP 57092; FR 2334659; US 4072698
IC **C07B055-00**; C07C231-20; C07C237-20
ICM **C07B055-00**; C07C229-06; C07C231-16; C07C237-04; C07C237-20
ICS C07C023-20; C07C231-20; C07C237-02; C07C237-12; C07C319-20; C07C323-59; C07C323-60
AB EP 442585 A UPAB: 19931220
Process for **racemisation** of optically active amino acid amides or **Schiff** bases thereof, comprising (a) reacting an amino acid amide with a carboxylic acid in the presence of a solvent and an aldehyde (b) recovering the salt of the **racemised** amino acid amide and the carboxylic acid. Water is added to the reaction mixt. in an amt. at least equivalent to the amt. of amide and the amt. of aldehyde is 0.5-4 equivs. w.r.t. the amide.

The amino acid amide is pref. phenylglycine amide, alanine amide, metionine amide or o-chorophenylglycine amide. Water is added at the beginning of the reaction and the reaction is at 75-100 deg.C. The amt. of water added is 0.5-3 equivs. w.r.t. the amt. of amide and the amt. of aldehyde is 1-2 is 0.5-3 equivs. w.r.t. the amt. of amide.

ADVANTAGE - The process gives high yields with fast **racemisation**. @(10pp Dwg.No.0/0)
FS CPI
FA AB; DCN
MC CPI: B10-B02F; E10-B02D1; E10-B02D6; E10-B02D8; E11-J
ABEQ EP 442585 B UPAB: 19940831
Process for the **racemization** of an optically active amino acid amide by reacting the amino acid amide with a carboxylic acid in the presence of a solvent and an aldehyde, characterised in that water is added to the reaction mixture and that the quantity of aldehyde amounts to 0.5-4 equivalents relative to the quantity of amino acid amide. Dwg.0/0

L71 ANSWER 5 OF 10 WPIX (C) 2002 THOMSON DERWENT
AN 1991-247268 [34] WPIX
DNC C1991-107299
TI Optically active aminoacid amide(s) prepn. - by reaction of aminoacid amide with carboxylic acid in presence of aldehyde and water.
DC B05 E14
IN BOESTEN, W H; BOESTEN, W H J
PA (STAM) STAMICARBON BV; (STAM) DSM NV
CYC 20
PI EP 442584 A 19910821 (199134)*
R: AT BE CH DE ES FR GB GR IT LI NL SE
NL 9000386 A 19910916 (199140)
HU 56532 T 19910930 (199143)
CS 9100418 A 19910915 (199148)
JP 05178805 A 19930720 (199333) 10p C07C237-06
EP 442584 B1 19931110 (199345) EN 14p C07C231-20
R: AT BE CH DE DK ES FR GB GR IT LI NL SE
TW 211555 A 19930821 (199347) C07B057-00
DE 69100598 E 19931216 (199351) C07C231-20
US 5306826 A 19940426 (199416) 8p C07C231-20
ES 2062660 T3 19941216 (199505) C07C231-20
CZ 281203 B6 19960717 (199637) C07C231-16
HU 212703 B 19961028 (199702) C07B055-00 <--
JP 2854148 B2 19990203 (199910) 10p C07C237-06
KR 179028 B1 19990515 (200052) C07C231-20
ADT EP 442584 A EP 1991-200306 19910214; NL 9000386 A NL 1990-386 19900216; JP 05178805 A JP 1991-22137 19910215; EP 442584 B1 EP 1991-200306 19910214; TW 211555 A TW 1991-101328 19910221; DE 69100598 E DE 1991-600598 19910214, EP 1991-200306 19910214; US 5306826 A US 1991-655623 19910215; ES 2062660 T3 EP 1991-200306 19910214; CZ 281203 B6 CS 1991-418 19910218; HU 212703 B HU 1991-482 19910213; JP 2854148 B2 JP 1991-22137 19910215; KR 179028 B1 KR 1991-2589 19910213
FDT DE 69100598 E Based on EP 442584; ES 2062660 T3 Based on EP 442584; CZ 281203 B6 Previous Publ. CS 9100418; HU 212703 B Previous Publ. HU 56532; JP 2854148 B2 Previous Publ. JP 05178805
PRAI NL 1990-386 19900216
REP EP 1821; EP 7834; FR 2173232; FR 2334658; US 4072698
IC **C07B055-00**; C07B057-00; C07C231-20; C07C237-20; C07C249-02
ICM **C07B055-00**; C07B057-00; C07C231-16; C07C231-20; C07C237-06
ICS C07C231-22; C07C237-02; C07C237-18; C07C237-20; C07C249-02
AB EP 442584 A UPAB: 19930928
Process for prepn. of optically active amino acid amides, characterised by adding water to the mixt. and comprising (a) at least partial conversion of a mixt. of L-amino and D-amino acid amides in a suitable solvent and the presence of an aldehyde and an optically active carboxylic acid to the corresp. amino acid amide and carboxylic acid salt (b) sepn. of a portion contg. mainly one of the diastereo isomers of the salt from the reaction mixt. The amt. of aldehyde is 0.5-4 equivs. w.r.t. the amt. of amino acid amide. The L-amino and D-amino acid amides are opt. the corresp. **Schiff** bases. The prod. is opt. treated with a mineral acid before sepn.

Specifically claimed are LD or DL salts of phenylglycine amide and mandelic acid; p-hydroxyphenylglycine amide and mandelic acid; methionine amide and 2-pyrrolidone-5-carboxylic acid; homophenylalanine amide and L-Z-aspartic acid. The amino acid amide is pref. phenylglycine amide or p-hydroxyphenylglycine amide and the carboxylic acid is L- or D-mandelic acid or 2-pyrrolidone-5-carboxylic acid.

USE/ADVANTAGE - The process is useful for the prepn. of amino acids. E.g. 99.8% optically pure prods. are obtd. with 99% efficiency.

0/0
FS CPI
FA AB; DCN

MC CPI: B07-D03; B10-B02F; E07-D03; E10-B02D; E10-C04D4
ABEQ EP 442584 B UPAB: 19931220
Process for the preparation of optically active amino acid amide whereby a mixture of the L-amino and D-amino acid amides in a suitable solvent in the presence of an aldehyde is converted in whole or in part, by means of an optically active carboxylic acid, into the salt of the amino acid amide and the carboxylic acid, and a portion mainly consisting of one of the diastereoisomers of that salt is separated from the reaction mixture obtained, characterised in that water is added to the reaction mixture and that the quantity of aldehyde amounts to 0.5-4 equivalents relative to the quantity of amino acid amide, and that the temperature during the conversion is between 70 and 120 deg.C.
Dwg.0/0
ABEQ US 5306826 A UPAB: 19940608
Prepn. of an optically active aminoacid amide comprises (a) (1)mixing together mixt. of corresp. **Schiff** bases of L- and D-aminoacid amides selected from gp. phenylglycine-,p-hydroxyphenylglycine-, methionine- and homophenylalanine-amides, a solvent, an optically active carboxylic acid selected from gp. mandelic, 2-pyrrolidone-5-, and Z-aspartic acids, and 1(+) equiv., of water w.r.t. **Schiff** base to produce the salt of the aminoacid amide and the carboxylic acid.
Alternatively, (2) mixing mixt. of the above L- and D- aminoacid amides with 0.5-4(1) equivs. of an aldehyde, a solvent and water to form the above salt; (b) one of the diastereoisomers of the salt is then sepd. and converted into the corresp. aminoacid amide.
Pref. pressure is 0.01-1 MPa and temp. 70-120 (75-100) deg.C for 1-8 hrs.. The salt may be treated with mineral acid before sepn..
ADVANTAGE - High yields of optically active aminoacid amide or corresp. aminoacid are rapidly obtd..
Dwg.0/0

L71 ANSWER 6 OF 10 WPIX (C) 2002 THOMSON DERWENT
AN 1991-046443 [07] WPIX
DNC C1991-019607
TI **Racemisation** of optically active halo-aryl-alkylamine(s) - by halogenating to N-halo cpds., dehydrohalogenation, and redn. of **Schiff** bases formed.
DC B05
IN KISS, G; MOZSOLITS, K; TAKACS, K; TOROK, Z
PA (CHIN) CHINOIN GYOGYSZER ES VEGYESZETI
CYC 1
PI HU 53853 T 19901228 (199107)*
ADT HU 53853 T HU 1989-330 19890126
PRAI HU 1989-330 19890126
IC **C07B055-00**
AB HU 53853 T UPAB: 19930928
Optically active (halo-aryl)-alkyl-amines of general formula (I) (where R = tri:halo-methyl gp.; R1 and R2 are independently hydrogen atom or 1-5C straight or branched alkyl gps.) are **racemised** by converting them to N-halo cpds. using a halogenating agent of formula (II) (where X = chlorine or bromine atom). The N-halogen cpds. are dehydro-halogenated to **Schiff** bases of formula (III), which on redn. yield **racemic** cpds. of formula (I).
FS CPI
FA AB
MC CPI: B10-B04B

L71 ANSWER 7 OF 10 WPIX (C) 2002 THOMSON DERWENT
AN 1991-046442 [07] WPIX
DNC C1991-019606
TI **Racemisation** of optically active tri-halo-methyl -aryl-alkylamine(s) - by halogenation and redn. of the active cpd..
DC B05

IN AJZERT, I; ECSERYNE, P; HERMECZ, I; KISS, G; MOZSOLITS, K; SZINNYEI, E; TAKACS, K
PA (CHIN) CHINOIN GYOGYSZER ES VEGYESZETI
CYC 1
PI HU 53852 T 19901228 (199107)*
ADT HU 53852 T HU 1989-327 19890126
PRAI HU 1989-327 19890126
IC **C07B055-00**
AB HU 53852 T UPAB: 19930928
Racemisation of optically active tri:halo-methyl)-aryl)-alkyl-amines of general formula (I), (where R = tri:halo-methyl gp. and R1 and R2 = independently hydrogen atoms or 1-5C straight or branched alkyl gp.) takes place, when the optically active cpd. (I) is treated by a halogenating agent of formula (II) (where X = chlorine or bromine atom) to yield a **Schiff** base (III). This base yields a **racemic** cpd. (I), on redn..
FS CPI
FA AB
MC CPI: B10-B04B

L71 ANSWER 8 OF 10 WPIX (C) 2002 THOMSON DERWENT
AN 1991-009425 [02] WPIX
DNC C1991-004139
TI Synthesis, inversion, and de-**racemisation** of asymmetric cpds. - comprises grafting reactant esp. aminoacid, onto polymer contg. chiral gps., treating the graft and then hydrolysing.
DC A14 A89 B05 E19 J04
IN CALMES, M; DAUNIS, J; JACQUIER, R
PA (CNRS) CNRS CENT NAT RECH SCI; (RHON) RHONE-POULENC CHIMI; (RHOD) RHODIA CHIM; (RHON) RHONE POULENC CHIM
CYC 25
PI EP 406124 A 19910102 (199102)* 10p
R: AT BE CH DE ES FR GB GR IT LI LU NL SE
WO 9100303 A 19910110 (199105)
W: AU BR CA FI HU JP KP KR NO RO US
FR 2649098 A 19910104 (199109)
AU 9059679 A 19910117 (199117)
US 5280093 A 19940118 (199404) 7p C08F226-00
US 5281750 A 19940125 (199405) 12p C07B057-00
EP 406124 B1 19991124 (199954) FR C08F220-58
R: AT BE CH DE DK ES FR GB GR IT LI LU NL SE
DE 69033361 E 19991230 (200007) C08F220-58
ES 2138580 T3 20000116 (200011) C08F220-58
ADT EP 406124 A EP 1990-401899 19900629; FR 2649098 A FR 1989-8679 19890629; US 5280093 A Cont of US 1990-545526 19900629, Cont of US 1992-915758 19920721, US 1993-47001 19930414; US 5281750 A CIP of US 1990-545526 19900629, Cont of US 1990-636476 19901231, US 1992-976672 19921116; EP 406124 B1 EP 1990-401899 19900629; DE 69033361 E DE 1990-633361 19900629, EP 1990-401899 19900629; ES 2138580 T3 EP 1990-401899 19900629
FDT DE 69033361 E Based on EP 406124; ES 2138580 T3 Based on EP 406124
PRAI FR 1989-8679 19890629
REP 1.Jnl.Ref; EP 300448; FR 2515645
IC C07B053-00; C07B057-00; C07C229-00; C08F220-58; C08F246-00; C09K019-38
ICM C07B057-00; C08F220-58; C08F226-00
ICS C07B053-00; **C07B055-00**; C07C227-30; C07C229-00; C08F220-36; C08F246-00; C09K019-38
AB EP 406124 A UPAB: 19930928
Process comprises (A) assymetric synthesis, (B) configuration inversion, and (C) **deracemisation**, involving grafting of a reactant onto a polymer (I) (itself also claimed) contg. blocks of chiral units (pref. 50-75%, most pref. 75%), functionalisation units, and opt. crosslinking units. Processes (B) and (C) involve treatment of the graft with a **racemising** or inverting reactant.

Synthesis of H2-N-C(R1)(R3)-(CH2)-COOH (II), (where R1 = H, alkyl or aralkyl; R3 = alkyl or aralkyl, but not = R1; n = 0 or 1), comprises (a) reversibly grafting H2N-C(R1)H-(CH2)n-COOR2 (III) (where R2 = 1-5C alkyl or aryl) onto (I) by forming a **Schiff**'s base; (b) deprotonating (III) with a strong base in an aprotic solvent (pref. THF) at ambient temp. (or pref. at the reflux temp. of THF for 15-240 mins.); (c) alkylating (esp. using R3X, where X = Cl, Br or I), or protonating (esp. with water, alcohol, mineral acid or organic acid) to create an assymetric C atom; (d) hydrolysing this **Schiff**'s base to yield (II).

ADVANTAGE - The process is highly selective in producing a single enantiomer. (10op Dwg.No.0/0)

FS CPI
FA AB; DCN
MC CPI: A12-W11L; B04-C03; B10-B02; B11-B; E10-B02B; J04-X
ABEQ US 5280093 A UPAB: 19940307

Polymers obtd. by free radical co-polymerisation of chiral unit(s), and functionising unit(s) having a protective function are new. Each chiral unit is a chiral monomer from one of two stereoisomers, (R and S), having a chiral C and M.W. not above 200, and possessing a double bond for polymerisation spaced at up to 5 (3 or 2) atoms from the chiral C. The chiral unit represents at least 1/2 (3/4) the mole units in the polymer, and if two or more chiral units are copolymerised with the functionalising agent all chiral units are of the same configuration, R or S.

Functionalising agents comprise an aromatic aldehyde, with the chiral monomer and a protective gp. Provided that the chiral monomer is not 1-acryloyl-2-methoxy methylpyrrolidine. Polymers opt. include crosslinking agents. Pref. one functional gp. is capable of hydrogen bonding to a 2nd identical chiral unit, and may be acidic, alcohol amide or amine. (benzaldehyde or aminobenzaldehyde). Typically the chiral monomer is (R)- or (S)-N-acryloyl-prolinol with functionalising agent para-(N-acryloyl-N-methylamino) benzaldehyde, free of methacryloyl. Pref. crosslinking agent is bis(acryloyl)-N,N-; dimethylethylenediamine or bis (acryloyl)-piperidine, with any acryloyl opt. replaced by methacryloyl.

USE - Chiral organic assymetric synthesis of pure enantiomers of amino acids and for changing from one enantiomer to another (**deracemising**). Using these supports synthesis may be done easily at R.T. (or higher), with yields 96-98%.

Dwg.0/0

ABEQ US 5281750 A UPAB: 19940315

Asymmetric synthesis comprises reversibly reacting a prochiral deriv. or enantiomer(s) with a functionalising unit of a support polymerised or copolymerised with chiral unit(s) which may also be a source of the functionalising unit or copolymerised from chiral and functionalising unit(s). The prochiral part of the reacted prochiral deriv. or enantiomer is then converted into a species having a reactive achiral portion. Thermodynamic equilibrium is attained at at least 20 deg. C. giving an asymmetric C atom from the achiral portion of the species and a 2nd species contg. this asymmetric C atom present in 85+(99+) % enantiomeric excess is sepd. from the support. The chiral and functionalising units may be copolymerised in presence of a crosslinking agent. Typically the prochiral deriv. is of formula H2N-C(R1)H(CH2)nCOOR2 (where n is 0 or 1; R1 is H and R2 is 1-5C alkyl o aryl). E.g. the chiral unit is N-acryloylprolinol, prolinolmethyl ether or prolinol and is in R or S form.

USE - Used for asymmetric synthesis, **deracemisation** and optical inversion of organic chiral cpds. The asymmetric synthesis of aminoacids, esp. of formula H2N-C(R1)(R3)-(CH2)nCOOH (where R3 is alkyl or aralkyl), the 2nd species contg. the asymmetric C atom being sepd. from the support by hydrolytic cleavage of the connecting bond.

Dwg.0/0

L71 ANSWER 9 OF 10 WPIX (C) 2002 THOMSON DERWENT
AN **1966-32423F** [00] WPIX

TI **Racemisation** of optically active amino acids.
DC B00
PA (AJIN) AJINOMOTO KK
CYC 2
PI FR 1517674 A (196800)*
FR 194 M (196801)
CA 854295 A (197043)
PRAI JP 1962-2811 19620131
AB FR 1517674 A UPAB: 19930831
Process for the **racemisation** of optically active amino acids by heating with a **racemisation** catalyst comprising a **metallic** ion
and a water-insoluble resin containing benzene or heterocyclic groups substd. by CHO with a group in the ortho position allowing chelation of the **metallic** ion.
Racemisation of unwanted forms of optically active amino acids partic. those arising from resolutions.
The resin is prepd. (a) by polymerisation of monomers contng. the chelating groups, the CHO groups being protected as **Schiff** bases or acetals, and (b) by suitably polymerising o-cresol with formalin and oxidising the methyl to CHO. The **metal** ions used are derived from Cu, Al, Fe, Zn, etc. An aqs. soln. of the amino acid at pH >8 and pref 10 is passed over the catalyst at >80 deg. and pref. 100 deg. alpha-amino-acids such as glutamic acid, valine, arginine, phenylalanine, aspartic acid and methionine may be **racemised** in yields up to 100%.
FS CPI
FA AB
MC CPI: B04-C02; B04-C03; B05-A01B; B05-A03; B10-A17; B10-B02B; B11-B; B11-C

L71 ANSWER 10 OF 10 WPIX (C) 2002 THOMSON DERWENT
AN **1966-29344F** [00] WPIX
TI **Racemizing** optical active amino acid.
DC B00
PA (TOAG) TOA GOSEI CHEM IND LTD
CYC 1
PI JP 42011924 B (196800)*
PRAI JP 1965-29557 19650521
AB JP 67011924 B UPAB: 19930831
Racemisation of optically active amino acids.
Process may be applied to a pharmacologically inactive optical isomer to convert it to the pharmacologically active **racemate** e.g. D-methionine to DL-methionine.
The amino acid is mixed with 5-30 mol.% of a salt of oxalacetic acid (I) together with **metal** ions e.g. of Cu, Fe, Al or Ni at pH 3-10, pref. 4-7 in aqueous/alcoholic solution at 50-140 deg.C, pref. 80-110 deg.C. The reaction scheme is as follows:
The **metal** ions form a chelate with the **Schiffs** base and
increase the effect of **racemisation**.
FS CPI
FA AB
MC CPI: B10-B01B; B10-B02B; B10-C02; B11-C; B12-J01

=> d his

(FILE 'HOME' ENTERED AT 12:06:11 ON 08 JUL 2002)
SET COST OFF

FILE 'HCAPLUS' ENTERED AT 12:06:23 ON 08 JUL 2002
E HOF R/AU

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L1          92 S E3,E6,E9-E11,E13
               E HERMSEN P/AU
L2           2 S E4,E5
               E DE BODE R/AU
L3           7 S E3,E4
               E DEBODE R/AU
               E DSM/PA,CS
L4        3490 S E3,E4
L5          99 S L1-L3
               E RACEMIZATION/CT
               E E3+ALL
L6        2539 S E4
               E E7+ALL
L7         984 S E5,E4
               E RACEMIZATION/CT
               E E7+ALL
L8         217 S E4,E5,E3
               E RACEMIZATION/CT
               E RACEMIZATION/CW
L9        3112 S E3
L10          1 S L5 AND L6-L9
L11          2 S L4 AND L6-L9
L12          3 S L10,L11
L13          5 S L5 AND RACEMI?
L14          5 S L10,L13
               E SCHIFF/CT
               E E19+ALL
L15       8039 S E5
L16      11132 S E5+NT
L17         77 S SHIFF?(L)BASE
L18      24674 S SCHIFF?(L)BASE
L19        541 S SCHIFF?(L)BASIC
L20         39 S L15-L19 AND L6-L9
L21        297 S L15-L19 AND RACEMI?
L22        297 S L20,L21
L23         72 S L22 AND ENANTIOM?
L24        110 S L22 AND (AMINOACID OR AMINO ACID OR PROTEIN OR ?PEPTIDE?)
L25         81 S L22 AND (AMINO ACID? OR PROTEIN? OR PEPTIDE?)/SC,SX
L26        124 S L24,L25
L27         79 S L22 AND ENANTIO?
L28         41 S L23,L27 AND L26
               E BASE/CT
               E E66+ALL
L29          1 S E1+NT AND L26
               E BASES/CT
L30          1 S L26 AND (E20 OR E22 OR E23 OR E24)
L31          5 S METAL(L)(ALKOXIDE OR ALKYL OR AMIDE OR HYDRIDE) AND L22
               E METAL ALKOXIDE/CT
               E E4+ALL
L32      16304 S E3,E4,E2+NT
               E METAL ALKYL/CT
               E E47+ALL
L33      26589 S E2+NT
L34          2 S L32,L33 AND L22
L35          6 S L29-L31,L34
L36          1 S L14 AND L15-L35
L37          6 S L35,L36
               SEL DN AN 2 6
L38          4 S L37 NOT E1-E6
L39         39 S L28 NOT L35-L38
L40         23 S L39 AND 34/SC
L41         16 S L39 NOT L40
               E AMIDES/CT
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L42          11 S L22 AND (AMIDE# OR AMINE#)/CW
                SEL DN AN  1 4 5 6
L43           4 S L42 AND E1-E12
L44           4 S L37 NOT (LIGAND OR COMPLEX)/TI
L45           6 S L43,L44
L46           6 S L45 AND L1-L45
L47       44619 S L6-L9 OR ?RACEM?
L48         341 S L47 AND (SHIFF OR SCHIFF)(L)(BASE OR BASIC?)
L49           2 S L48 AND L32,L33
L50           8 S L48 AND METAL?(L)(ALKOXIDE OR ALKYL OR AMIDE OR HYDRIDE)
L51           9 S L49,L50
L52          11 S L46,L51
L53           8 S L52 NOT (LIGAND OR COMPLEX)/TI
L54           8 S L53 AND L1-L53
L55           4 S L24,L25 AND L54
L56           8 S L54,L55

     FILE 'HCAPLUS' ENTERED AT 12:35:02 ON 08 JUL 2002

     FILE 'REGISTRY' ENTERED AT 12:38:21 ON 08 JUL 2002
L57           1 S 865-47-4
L58           1 S 381724-98-7
L59           1 S 381724-99-8
L60           3 S C13H18N2O/MF AND BUTANAMIDE AND 46.150.18/RID

     FILE 'HCAPLUS' ENTERED AT 12:39:51 ON 08 JUL 2002
L61           1 S L58 OR L59

     FILE 'REGISTRY' ENTERED AT 12:40:17 ON 08 JUL 2002

     FILE 'WPIX' ENTERED AT 12:40:41 ON 08 JUL 2002
                E EP1167347/PN
L62           1 S E3
L63         325 S C07B055/IC,ICM,ICS,ICA,ICI
L64           7 S L63 AND (SCHIFF? OR SHIFF?)
L65           7 S L62,L64
L66          37 S ?RACEM? AND (SCHIFF? OR SHIFF?)
L67          31 S L66 NOT L65
L68           9 S L67 AND ?METAL?
                SEL DN AN 8 9
L69           2 S L68 AND E1-E2
                SEL DN AN L67 11
L70           1 S E3-E4
L71          10 S L65,L69,L70 AND L62-L70

     FILE 'WPIX' ENTERED AT 12:53:36 ON 08 JUL 2002

     FILE 'DPCI' ENTERED AT 12:54:12 ON 08 JUL 2002
                E EP1167347/PN
                E NL1015495/PN
                E US2001056209/PN
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